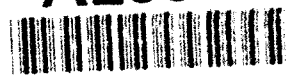

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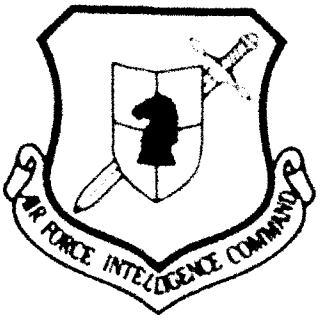
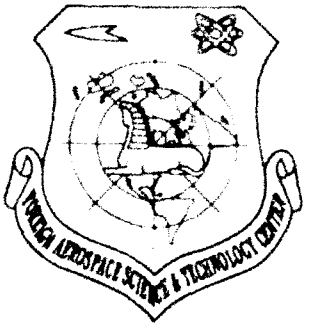


ANELASTIC RELAXATIONS ASSOCIATED WITH LOCAL DISORDERING IN GRAIN BOUNDARIES

by

Cheng Bolin, Ge Tingsui



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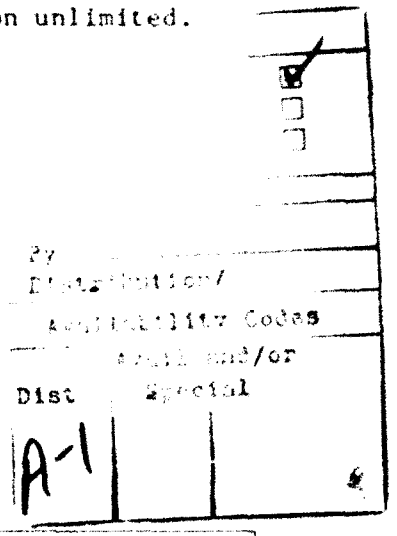

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Anelastic relaxations associated with local disordering in grain boundaries

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Abstract: Internal friction and micro-creep measurements were performed with high-purity Al bamboo-crystal specimens. The relaxation strength was found to decrease with the decrease of the temperature of measurement and became zero at about $0.4T_m$ (T_m being melting temperature). This reflects the occurrence of local disordering in the bamboo boundary region at this temperature. This result conforms to the picture of grain boundary disordering constructed by atomic simulation studies.

Key Words: grain boundary disordering, internal friction, micro-creep, high purity Al.

The structural stability of the metal grain boundary is a long debated problem. The issue being debated is whether the grain boundary can be melted to a structure similar to that of the super cooled liquid at a temperature far below the melting point T_m of the material. Using the lattice gas model, Kikuchi and Cahn[1] studied the two dimensional symmetric SS inclined boundary. They discovered that disordering occurs on the boundary when heated to a temperature approximately $0.5T_m$, and finally at T_m "complete" melting occurs.

However, contrary to the above conclusion, calculations using molecular dynamics by Kalonji et al.[2] shew that the boundary

underwent first order phase transition at the temperature of $0.8T_m$. The grain boundary of the crystal was replaced by disordered layer similar to a liquid. Simulations by Nguyen and Yip[3] also indicated that the ordering of the grain boundary was completely lost within the temperature of 0.8-0.9 T_m.

Using electron microscope, Glicksman and Vold[4] observed that the grain boundary of Bi was replaced by a layer similar to a liquid at a temperature far below the melting temperature. On the other hand, using electron microscopes, Balluffi et al.[5] observed that even when the temperature of Al was raised to 0.999 T_m, no trace of melting was seen.

This paper attempts to further explain the above controversy through a series of anelastic relaxation experiments, especially to achieve understanding about the grain boundary disordering phenomenon before the complete melting. The results of the fine grain Al has been reported previously[6]. This paper stresses on the results of anelastic measurement of the bamboo-crystal boundary and compares them with the results for the fine grain crystals.

1. Experimental procedure

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The specimens used in the experiment are 99.999% and 99.9999% pure Al. The initial state of 99.9999% Al is that the fine thread is obtained by heat extrusion at 570-640K, and the crystal grains have experienced some growth from metal phase observation; some grain size has exceeded the diameter of the specimen. The 99.999% Al is cut into strips with dimensions 2 ¥ 2 ¥ 135mm; the strips are then cold extended to fine thread of 1mm diameter; the area contraction is 84%RA.

To prepare the bamboo-crystal specimen, one of the following three methods is used: (1) high temperature static annealing, (2) high temperature static annealing after predeformation, (3) high

temperature dynamic annealing after predeformation. To obtain the fine grain specimen, only the standard static annealing is needed.

The anelastic experiments described in this paper consists of micro-creep and internal friction. The instrument used for the measurement is a homemade multi function internal friction meter. The entire apparatus is controlled by an IBD-PC computer with 8087 coprocessor, which also conducts real time data processing. During forced vibration, the frequencies can be varies from 10^{-5} to 10 Hz. The resolution of the internal friction measurement is 1 ¥ 10^{-4}.

During the experiment, the measurement of the internal friction and the modulus is done by lowering the temperature. The internal friction is measured by forced vibration method; the maximum amplitude of the strain is 1 ¥ 10^{-5}. The creep measurement is done by stepwise temperature increasing method; the elastic strain is 1 ¥ 10^{-4}. The rise time of the loading is 0.2s. afterwards, constant stress is maintained.

2. Experimental results

2.1 The relaxation strength of the bamboo-crystal specimen

Fig. 1 gives the internal friction results of the 99.999% Al specimen obtained by forced vibration with varying frequencies. The specimen is 48 mm long and has the bamboo-crystal boundary number N =22. The curves in the figure are obtained after 2 hours annealing at 873K. After subtracting the high temperature index background, corresponding to each curve, the peak to peak temperatures T_p of the internal friction are 478, 443, 418K respectively. The heights of the peaks Q_m^{-1} are 0.081, 0.078, 0.074 respectively. The half height widths of the peaks are 4.6 ¥ 10^{-4}, 4.9 ¥ 10^{-4}, 5.3 ¥ $10^{-4}K^{-1}$.

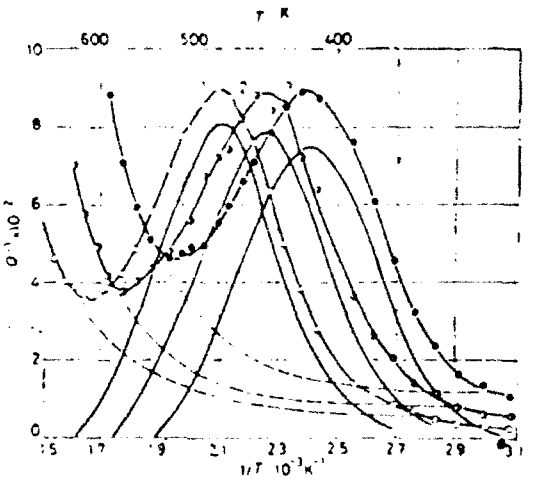

Fig. 1 Variation of bamboo boundary internal friction peak with frequency in 99.999%Al
1—○—1Hz; 2—◑—0.1Hz; 3—●—0.01Hz
——with internal friction background subtracted

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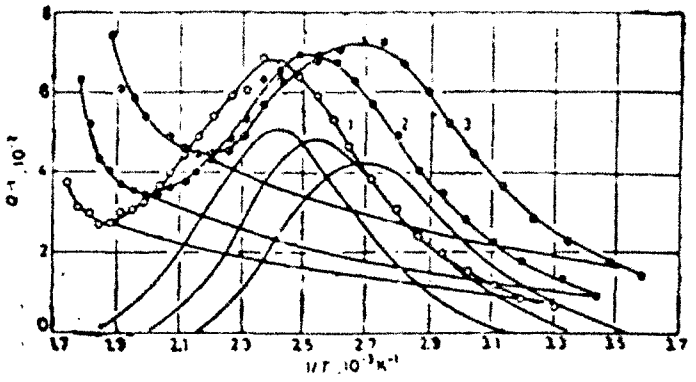

图 2 99.9999%Al中竹节晶界内耗随测量频率的变化

Fig. 2 Variation of bamboo boundary internal friction peak with frequency in 99.9999%Al

1—○—1Hz; 2—●—0.1Hz; 3—◑—0.01Hz

——with internal friction background subtracted

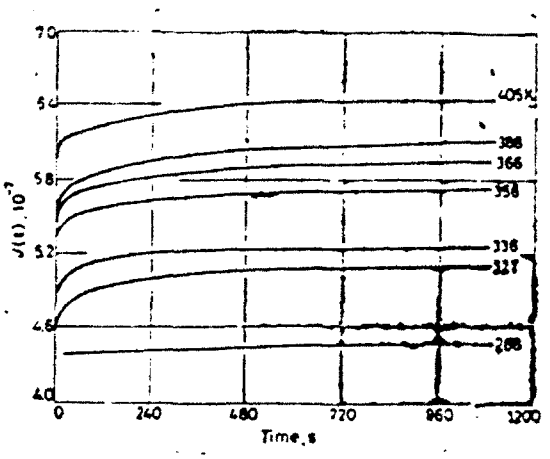

图 3 在不同温度下99.9999%Al竹节晶的蠕变曲线

Fig. 3 Creep curves of 99.9999% Al bamboo-crystal specimen at various temperatures

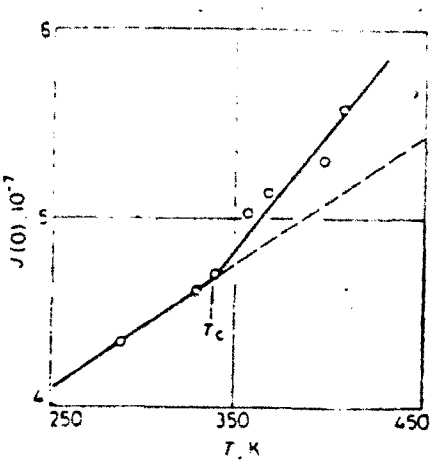

图 4 在不同温度下99.9999%Al竹节晶的初始顺度$J(0)$

Fig. 4 Initial compliance $J(0)$ of 99.9999%Al bamboo-crystal specimen at various temperatures

T_c —temperature of experimental curve starting to deviate from straight line

Fig. 2 gives the internal friction curve of the 99.9999% Al bamboo-crystal specimen. The specimen is 58mm long and the boundary number of the bamboo-crystal is N = 17. After 2 hours of annealing

at 873K, temperature lowering measurement is done. After subtracting the high temperature index background, the T_p values are 412, 391 and 368 and the Q_m^{-1} values are 0.050,0.048, 0.042 respectively. The half-height widths of the peaks are respectively 5.50 ¥ 10^{-4}, 5.65 ¥ 10^{-4}, 6.30 ¥ 10^{-4} K^{-1}. From Fig. 1 and Fig. 2, we conclude that the peak height of the internal friction decreases as the peak temperature decreases, i.e. the boundary relaxation strength is a quantity which depends on the temperature.

To obtain quantitative relationship between the relaxation strength and the temperature, micro-creep measurement of the specimen is conducted. Fig. 3 shows the creep curve of the above described 99.9999% Al bamboo-crystal specimen. When the measurement temperature is 288K, no noticeable creep is observed during the time scope of observation. To accurately calculate the initial compliance J(0), we have also plotted the experimental curve in Fig. 3 as the curve of J(t) corresponding to lg t. Due to the magnifying effect in short time interval of lg t, from such a curve we can very accurately calculate J(0).

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表 1 99.9999%Al中竹节晶界弛豫强度Δ与温度T的关系

Table 1 Relaxation strength, Δ, and temperature, T, in 99.9999% Al bamboo boundaries

T, K	327	336	346	356	366	386	405
J_u (10^{-3})	4.60	4.65	4.74	4.82	4.88	5.00	5.16
Δ	0.104	0.125	0.171	0.191	0.213	0.220	0.245

表 2 99.9999%Al中竹节晶界弛豫的热激活参量

Table 2 Activation parameters of bamboo boundary relaxation in 99.9999%Al

T_p, K	Q_m^{-1} (10^{-4})	β	Δ
412	50	4.95	0.36
391	48	5.13	0.35
368	42	5.75	0.31

H=1.37 eV; τ_1=3.0×10^{-15}s

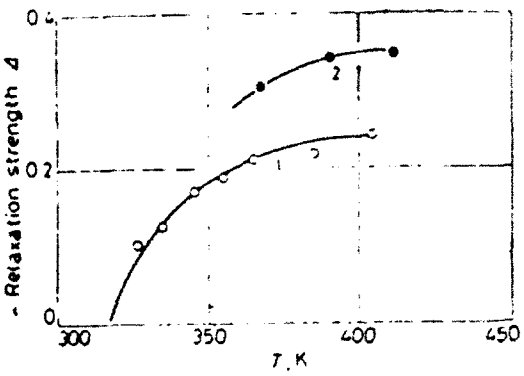

图 5 99.9999%Al竹节晶弛豫强度随温度的变化

Fig. 5 Variation of relaxation strength Δ with temperature T for bamboo boundary relaxation in 99.9999%Al

1—○—from creep measurement;

2—●—from internal friction measurement

Experimental and theoretical work proves that if no relaxation phenomenon occurs in the single crystal of the metal, its elastic modulus varies as a linear function of the temperature on a large temperature range[7]. Consequently, if no creep occurs in the specimen, the unrelaxed compliance J_u [= J(0)], under different temperatures and with the same torque, should be a linear relationship of the temperature as the straight line part of Fig. 4 indicates. Fig. 4 shows that J_u deviates from the straight line at $T = T_c$, which indicates that at this temperature the stick-slip (stress relaxation) of the grain boundary becomes obvious. Using the bulk melting point T_m=933 as the unit, the temperature obtained from Fig. 4 is $T_c = 0.35T_m$.

Based on the creep formula $J(t) = J_u + dJ \cdot y(t)$, where y(t) is the normalized creep function, the grain boundary relaxation strength under different temperatures can be obtained. Its definition is $D=dJ/J_u$. From the curves in Fig. 3 and Fig. 4 we can obtain J_u and dJ and thus D (shown in Table 1). Fig. 5 gives the relationship between the relaxation strength and temperature T. From that we know that D has a parabolic relationship with T. When temperature decreases, D suddenly decreases and reaches zero at a temperature T_0. From Fig. 5 $T_0=0.34T_m$ is obtained, which is consistent with the T_c of Fig. 4.

2.2 The thermal excitation parameter of the bamboo-crystal grain boundary relaxation

If the bamboo-crystal grain boundary has a unique relaxation time, the peak height of the internal friction Q_m^{-1} should be related to the relaxation strength D according to $Q_m^{-1} = D/2$. However, the peak width shown in Fig. 2 indicates that there exists a distribution of the relaxation time. Using the treatment in Ref [8], listed in Table 2 are the b parameters of the internal friction peak for 99.9999% Al bamboo-crystal boundary indicated in Fig. 2. Hence we conclude that the b values in the Gauss distribution function of the relaxation time depend on temperature. Using linear fitting, we find that b and T obey the formula $b = |b_0$

$- b_H/kT|$, where b_0=1.83, b_H=0.24eV. This shows that the t_0 and H in the relaxation time relation $t = t_0\exp(H/kT)$ both have distribution and their variations are correlated. The negative sign in the above formula indicates that the variations of ln t_0 and H with the internal parameter are opposite. Experimentally measured values for 99.9999% Al are H = 1.37eV, t_0=3.0 ¥10^{-18}s.

It should be pointed out here that when the purity of the specimen increases, the distribution of the excitation energy b_H also increases. In the distribution of t, the distribution of H plays a more important role.

When there exists a distribution of the relaxation time, the internal friction can be described by

$$\tan f = \int f(J_2(x), J_1(x)),\ x = \ln wt.$$

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where the compliances $J_2(x)$, $J_1(x)$ are respectively[9]

$$J_2(x) = dJ \cdot f_2(x,b)$$

$$J_1(x) = J_U + dJ \cdot f_1(x,b).$$

Hence $$\tan f = \int f(dJ \cdot f_2(x,b), J_U + dJ \cdot f_1(x,b)).$$

At the peak, x =ln wt =0, $f_1(0, b)$=1/2. Therefore,

$$Q_m^{-1} = \int f(dJ \cdot f_2(0,b), J_U + dJ/2) = \int f(D \cdot f_2(0, b), 1+D/2).$$

The relaxation strength D (=dJ/J_U) thus obtain is

$$D = \int f(Q_m^{-1}, f_2(0, b) - Q_m^{-1}/2).$$

The resulting D values are listed in Table 2. They are also plotted in Fig. 5 (curve 2).

3. Discussion

In our experiments described above, we have observed for the first time the variation of the relaxation strength D of the grain boundary with the changes in temperature T. This indicates that the grain boundary anelastic relaxation is coupled with the temperature through the relaxation strength as well as the relaxation time.

The study of the thermodynamics of the relaxation phenomenon indicates that the relaxation strength is a measure of the degree

of coupling between the internal parameter x and the external stress. The change in x is related to the movement of the atoms at the grain boundary. At low temperature, the grain boundary has the stable low energy structure. When the temperature is raised, the structure of the grain boundary is damaged due to the thermodynamic movement of the atoms. According to CSL model of the grain boundaries, when the temperature rises, the number of the collapsing lattice points decreases (the point defects increases). This leads to the local disordering of the grain boundaries, which is the main idea of "disordered atomic model"[10]. Hence we believe that at the measured temperature T_c, disordering occurs in the local atomic structure of the grain boundaries.

Generally speaking, our results show that disordering can be noticeable at relatively low temperature ($\sim 0.4T_m$). However, this does not support the notion that the grain boundaries start melting.

The experiments conclude that the T_c for the bamboo-crystal grain boundaries ($0.34T_m$) is lower than that of the fine grain ($0.43T_m$). This in some degree reflects that the disordering in the bamboo-crystal grain boundaries is higher than in the fine grain. The values of grain slipping excitation energy also support this: H=1.3eV for 99.999% bamboo-crystal and H=1.5eV for fine grain crystal. Using electronic microscope, Berger et al.[12] have studied the recrystallization process of shape-deformed single crystal. They discovered that the grain boundaries formed at the early stage of the recrystallization are almost all of the collapsing position type. Regular grain boundaries occur later. Fontaine and Rocher[12] have reported that all boundaries of the fine grain multi-crystal Si can be described by the multi-twin relationship. The research of the relations between the grain boundary characteristics distribution of Al[13] and iron alloy[14] has shown that the occurrence of the collapsing grain boundaries decreases as the grain size increases. Hence our conclusion about the high density

of disordered grain boundaries in predeformed annealed bamboo-crystal is consistent with this research.

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The changes in relaxation strength can be explained as follows. Induced by stresses, the local disordering movement of bamboo-crystal boundaries is oriented, which causes slipping of the grain boundaries. The amount of slipping depends, on the one hand, on the available regions, on the other hand, on the factors restricting the slipping. We know that the intersection and interaction of the dislocation substructure near the bamboo-crystal boundaries constrains the slipping to be bounded[15]. The compliance of the restoring force provided is dJ. The relaxation strength is $D=dJ/J_u$. The stability of the dislocation substructure depends on the temperature. At low temperature, the state of dislocation can not easily relax and the dislocation substructure is stable. As a result the elastic restoring force provided is large, i.e. the compliance relaxation (dJ) is small. Hence the relaxation strength (D) is low. When the temperature is raised, the state of dislocation can relax, i.e. the compliance relaxation increases. Hence the relaxation strength increases. If the internal ordering parameter x is considered, dJ = cm, where the coefficient c indicates the coupling between the internal parameter and the strain, i.e. the stability of the dislocation substructure ($e^{an}=kx$). m is the equilibrium internal parameter value \|o(˙,x) corresponding to the unit stress s (\|o(˙,x)=ms). It signifies the local disordering of the grain boundaries and is a function of the temperature. Thus the fact the dJ increases as the temperature rises reflects not only the changes in the stability of the dislocation substructure, but also the changes in the degree of local disordering in the grain boundaries. Therefore we can use the relaxation strength as a measure of the degree of local disordering of the grain boundaries.

Reference

1 Kikuchi R, Cahn J W. Phys Rev, 1980; 21B: 1893
2 Kalonji G, Deymier P, Najafubadj R, Yip S. Surf Sci, 1984; 144: 77
3 Nguyen T, Yip S. Mater Sci Eng, 1989; 107: 15
4 Glicksman M E, Vold C L. Acta Metall, 1967; 15: 1409
5 Hsieh T E, Balluffi R W. Acta Metall, 1989; 37: 1637
6 Ke T S. Scr Metall, 1990; 24: 347
7 Ke T S. Phys Rev, 1949; 76: 579
8 Seeger A, Weller M, Diehl J, Pan Zhengliang, Zhang Jinxiu, Ke T S Z Metallk, 1982; 73: 1
9 Nowick A S, Berry B S, anelastic Relaxation in Crystalline Solids, New York: Academic Press, 1972; 94
10 Ke T S. J Appl Phys, 1949; 20: 274
11 Berger A, Wilbrandt P J, Haasen P. Acta Metall, 1983; 31: 1433
12 Fontaine C, Rocher A. J Microsc, 1980; 118: 105
13 Grabski M W. J Phys (Paris), Colloque, 1985; 46(C4): 567
14 Watanabe T. J Phys (Paris), Colloque, 1985; 46(C4): 555
15 Zhu A W, Ke T S. Phys Status Solidi (a), 1989; 113: 393

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